



PATENT
8007-1067

IN THE U.S. PATENT AND TRADEMARK OFFICE

In re application of

Tomio HAYASHI et al.

Conf. 1491

Application No. 10/500,935

Group 1755

Filed July 8, 2004

Examiner Carol Koslow

MAGNETITE PARTICLES

DECLARATION UNDER RULE 132

Assistant Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

Sir:

I, Tomio HAYASHI, am an employee of the current assignee, Mitsui Mining & Smelting Co., Ltd., Japan.

I have been engaged in researching and developing magnetite particles as a researcher in the Magnetic Materials Plant of Mitsui Mining & Smelting Co., Ltd., of Hibi, Okayama, Japan for 4 years and 9 months.

I am one of the inventors of the above-identified U.S. patent application. I am familiar with the Examiner's position that the recited coercive force of the claimed magnetite particles would have been obvious under 35 USC 103 (a) because the process conditions, particle sizes, and composition of the magnetite particles of U.S. patent No. 5,885,740 overlap the disclosed process conditions and claimed particle size and composition. However, the Examiner's position is incorrect.

The following experiments have been performed under my supervision to show that the magnetite particles comprising 0.1-1% phosphorus and an octahedral shape produced in accordance with US 5,885,740 actually have a lower coercive force than the recited coercive force of the claimed magnetite particles comprising 0.1-1% phosphorus and an octahedral shape. A comparison of the experimental data is provided in Table 1.

Experimental Procedure

Comparative experiment 1:

The experiment was carried out in accordance with Example 4 of US 5,885,740. Please be advised that only Example 4 produces magnetite having an octahedral shape, as recited in the claims of the present invention. Other Examples produce other shapes of magnetite. Thus, I carried out the experiment according to Example 4.

A reactor was preliminarily charged with 3.86 L of an aqueous NaOH solution (1.67 mol/L) containing 1.49 g of sodium hexametaphosphate (corresponding to 0.25 wt % as P with respect to Fe), 6.77 mL of a sodium aluminate solution (corresponding to 0.60 wt % as Al with respect to Fe) and 1.40 mL of a sodium silicate solution (corresponding to 0.15 wt % as Si with respect to Fe); the reactor was further charged with 4.27 L of an aqueous ferrous sulfate solution containing 1.50 mol/L of Fe^{2+} .

whereupon ferrous hydroxide formed (the use of sodium hydroxide corresponded to 1.15 equivalents with respect to Fe^{2+}).

The resulting ferrous hydroxide was heated at 90°C under mechanical agitation with air being supplied for 120 min at a flow rate of 2 L/min, thereby producing magnetite particles in accordance with Example 4 US 5,885,740. The resulting particles were washed with water, filtered, dried and comminuted by customary procedures.

Comparative experiment 2:

The same procedure was carried out as in comparative experiment 1 except that the amount of sodium hexamethaphosphate was charged to 3.97 g.

Comparative experiment 3:

The same procedure was carried out as in comparative experiment 1 except that the amount of sodium hexamethaphosphate was charged to 7.94 g.

Results and Discussions

The magnetite particles thus obtained were evaluated for properties and various characteristics in accordance with the same methods used to evaluate the claimed magnetite particles (see page 5 line to page 6 line 16 of the present specification), which are described below. The results are shown in Table I below.

Methods of evaluation:

(1) Phosphorus content

A sample dissolved in an acid was analyzed by ICP to determine the phosphorus content.

(2) Average particle size

The shape of particles was observed under a scanning electron microscope. A micrograph was taken at a magnification of 40,000 times, and the Feret's diameters of 200 particles were measured to calculate an average particle size.

(3) Specific surface area

Measured with a BET specific surface area analyzer, Micromeritics 2200 supplied by Shimadzu Corp, Japan.

(4) Saturation magnetization, residual magnetization, and coercive force

Measured with a vibrating sample magnetometer, VSM-P7 manufactured by Toei Industry Co., Ltd., Japan, in an external magnetic field of 796 kA/m and 79.6 kA/m.

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TABLE I

TABLE I												
	Avg. Particle Size (μm)	BET (m^2/g)	Magnetic Characteristics (796 kA/m)			Magnetic Characteristics (79.6 kA/m)				P content (mass %)	Shape	
			σ_s (Am^2/kg)	σ_z (Am^2/kg)	H_c (kA/m)	σ_s (Am^2/kg)	σ_z (Am^2/kg)	H_c (kA/m)				
Comparative Experiment 1	0.19	9.7	83.0	8.1	7.2	59.6	7.7	6.6	0.15	octahedral		
Comparative Experiment 2	0.19	9.0	82.0	9.2	6.3	56.5	8.4	7.4	0.30	octahedral		
Comparative Experiment 3	0.18	9.6	79.7	10.6	9.2	56.5	9.2	8.2	0.79	octahedral		

As is apparent from the above results, the magnetite particles produced in accordance with the cited reference showed lower coercive force (e.g. 7.2-9.2 kA/m in a magnetic field of 796 kA/m) than the claimed magnetite particles (e.g. 10-25 kA/m in a magnetic field of 796 kA/m in claim 1). It is considered that this is due to the difference in the production method of magnetite. In the present invention, the phosphorus compound is added to the reaction slurry during the oxidation reaction of Fe (see page 4, line 32 to page 5, line 7 and Examples 1 and 3 of the present specification). In contrast to this, in the cited reference the phosphorus compound is added to the reaction system prior to the oxidation of Fe (see column 4, lines 14-19 of the cited reference).

In view of the above, it can be concluded that the magnetite particles disclosed in the cited reference do not satisfy the claimed range of coercive force and therefore are not the same product of the claimed invention.

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I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States code and that such willful false Statements may jeopardize the validity of the application or any patent issuing thereon.

Tomio Hayashi
Tomio HAYASHI

Date Sep. 22, 2006